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NUCLEAR WASTE MANAGEMENT PROGRAM PROCEDURE

SP 12-3
CALIBRATION, USE, AND MAINTENANCE OF THE DELSA-440 ELECTROPHORETIC MOBILITY ANALYZER
Revision 0

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Author:	Nathalie Wall	*Original signed by Nathalie Wall*	*5/16/02*
	(printed name)	(signature)	date

1.0 Purpose and Scope

This procedure prescribes the Sandia National Laboratories (SNL) Nuclear Waste Management Program (NWMP) process for the verification of calibration, operation, maintenance of the DELSA (Doppler Electrophoretic Light Scattering Analyzer) 440 as part of the laboratory geochemistry research activities in support of the Waste Isolation Pilot Plant (WIPP) Project.

This procedure is applicable only for the DELSA 440. This document is not meant to substitute for the manufacturer's reference manual for the DELSA 440. The user is responsible for reading and understanding the manual (see references).

This document replaces TOP 540, Rev. 0. The only reason for this revision is to comply with SNL WIPP QA requirements.

Acronyms and definitions for terms used in this procedure may be found in the NWMP Glossary located at the Sandia National Laboratories (SNL) NWMP On-line Documents web site.

2.0 Implementation Actions

2.1 Safety

This document does not address ES&H issues. Laboratory ES&H procedures described in the SOPs of the laboratory in which the equipment is used shall be adhered to.

2.2 Responsibilities

The Principal Investigator (PI) or designee, whose activities warrant the use of this procedure is responsible for implementing the requirements of this procedure.

The Laboratory Technician, or designee, is responsible for performing the calibrations and measurements following the requirements of this procedure, documenting calibrations, and assuring that the latest revision of this document is followed.

2.3 Controls

Controls are established by written procedures or instructions prepared in accordance with NP 5-1, Implementing Procedures, of the Sandia National Laboratories WIPP Quality Assurance Program. Procedures are issued in accordance with NP 6-2, Document Control Process, of the Sandia National Laboratories WIPP Quality Assurance Program.

2.3.1 Standards

Calibration will be verified using commercially obtained electrophoretic mobility standards as QC samples. At this time, no national recognized standards (such as from NIST) are known to exist. The value of the commercially obtained standards will be verified by comparison to the measured value of a standard of independent origin. The manufacturers, lot numbers and expiration dates (if any) of the standards used shall be recorded in the laboratory notebook. The standards will not be used past the expiration date listed on the container by the manufacturer.

2.4 Quality Control

If results of a QC Sample are not within the control boundaries (see Section 2.4.2) of the electrophoretic mobility value of the standard, all samples bracketed by this QC sample shall be flagged on the data reports and corrective action documented with the data.

2.4.1 Calibration

There are no means for the user to calibrate the DELSA 440. The unit was factory-calibrated and its calibration was verified by the installing technician. Quality Control is implemented through use of performance tests.

2.4.2 Performance Test Criteria

Performance tests will be done by measuring the electrophoretic mobility of a QC sample, according to the instructions of the QC assay sheet. The measured mean mobility of the QC sample ($\pm$ 2σ) must fall within the limits specified on the QC sample assay sheet.

2.4.3 Corrective Action

If a performance test is failed, the cell position shall be checked to insure that the cell is positioned at the stationary layer (see section 2.4.5). If necessary, the cell should be repositioned and the test repeated. If it fails again, periodic maintenance as described in section 5 of the reference manual shall be implemented, and the performance test shall then be repeated. If the performance test is failed once again, troubleshooting as described on page 3-74 of the reference manual shall be consulted and corrective action taken as required. If the instrument still fails its performance test, it shall be tagged and taken out of service until repaired.

Failures of performance tests and the remedial action taken shall be documented on the analysis printout. Failures of more than one performance test in a given day shall be documented in the appropriate scientific notebook.

2.4.4 Frequency

The instrument's calibration shall be verified with performance tests immediately prior to and immediately after each day's use. If a set of analyses are done, a performance test will be done at least once every 10 analyses.

2.4.5 Stationary Layer

The stationary layer shall be located and the cell positioned in it prior to each day's analyses, as described on pages 3-29 through 3-33 of the reference manual.

2.4.6 Cell Constant

The cell constant shall be checked (as instructed on page 3-25 through 3-27 of the reference manual) annually, and whenever the cell has been disassembled or a new cell is being used.

2.4.7 Cell Preparation

The sample cell's optical surfaces shall be cleaned prior to each day's use. The operator must take care not to touch the optical surfaces, and should inspect to the cell prior to each analysis to ensure that these surfaces are free of dirt and moisture, and that no bubbles are in the optical path. The sample cell shall be flushed with at least 5ml of deionized water prior to filling with sample. When filling with sample, at least 2ml of sample shall be used. If cleaning of the cell is called for (by failure of a performance test), it should be cleaned in accordance with section 5.2 of the operator's manual. The cell should never be placed in an ultrasonic bath.

2.5 Procedure

Analyses will be performed as per instructions in section 3 of the reference manual.

2.5.1 Optimization

Analysis should not be initiated until the front and back cell temperatures are within 0.2°C of the "set cell" temperature (normally: 25°C).

For most samples, the following Run Parameters will be used:

- Use "current" mode, not voltage. Set current at about half the value of the conductivity.
- "Field on" time should be set at 2.0 seconds.
- "Field off" time should be set at 1.5 seconds.
- "Run time" should be set at about 80 seconds.
- The "Frequency" should be set at 500 Hz.
- If more resolution is desired, reduce the frequency.
- If more precision is desired, increase the run time.

If the above parameters are not applied, the instrument user will record in the laboratory notebook the new parameters.

2.5.2 Analysis

The software will list the electrophoretic mobilities (or zeta potentials) measured at each of the four angles if "Peak Analysis" is selected. However, these values are rarely in agreement. It is

recommended that the true value be determined through visual inspection of the peaks, using the following criteria, listed in descending order of importance:

1. Sharp peaks have more credibility than broad peaks.
2. Peaks in agreement (superimposed) with each other have more credibility than peaks that stand along.
3. High-angle (such as 34.7°) have greater credibility than low-angle (such as 8.7°) peaks.

Select "Frequency" under "View" on the run screen if multiple peaks are present to determine if any of the peaks shown are artifacts (see page 2.10 of the reference manual). NOTE: If the predominant peak has a value of zero, it is possible that it is an artifact that is caused by a bubble in the channel or dirt on the glass, and *may not* be identified as an artifact by this method. If any other peaks are present, check the chamber for bubbles, clean, and reanalyze.

Samples consisting of hydrophobic colloidal particles (e.g., mineral fragments) dispersed in moderate to high ionic strength electrolytes may be unstable, in that the colloidal particles are in the process of agglomerating. If the sample is though to be kinetically unstable, two sequential analysis, taken several minutes apart, should be made, and the results compared to see if the sample is stable.

The results may be recorded in the laboratory notebook in accordance with NP 20-2, Scientific Notebooks.

2.6 Maintenance

Maintenance will be performed on the instrument as instructed in section 5.1 of the reference manual. Sample cells should be flushed out with deionized water after each day's use.

2.7 References

- *Product Reference Manual – Coulter DELSA 440 Operating Instructions*, Oct. 1996 – PN 4235648B, pp.3-1 through 3-81 and pp. 5-1 through 5-16. (ERMS #522171)
- NP 5-1, Implementing Procedures
- NP 6-2, Document Control Process
- NP 20-2, Scientific Notebooks

3.0 Records

The following QA records, generated through implementation of this procedure, shall be prepared and submitted to the WIPP Records Center in accordance with NP 17-1 (Records):

QA Record	Preparer	Records Submitter
• Performance test and data printouts	Laboratory Technician or Designee	Principal Investigator or Designee
• Scientific Notebook	Laboratory Technician or Designee	Principal Investigator or Designee

4.0 Appendices

There are no appendices associated with this procedure.